

PREVENTION OF HYDROGEN EMBRITTLEMENT OF HIGH STRENGTH ALLOY STEEL USING SURFACE COATING TECHNIQUES

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ABSTRACT

High strength steel has been used for various engineering & industrial applications. Industries like Automobile, Aerospace, Power & Energy, Oil, Marine, Defence etc., were using high strength steel to develop new light weight energy efficient solutions with an improved efficiency and lower the overall weight of the structure. High strength steels are subjected to a perennial problem of hydrogen embrittlement during the course of manufacturing and under a specific condition of the operation resulting in an unexpected failure of components below the permissible stress limit and without any prior warning severely amputating the service life. This failure is predominantly brittle in nature & results in catastrophic accident. Seriousness of the issue demands a systematic analysis of parameters responsible for hydrogen embrittlement by simulating the failure in a controlled environment using several laboratory tests. EN24 steel, which is an alloy steel having nickel, chromium & molybdenum as main alloying elements offers good machinability and claims a high strength with the fine resilience, hardness and wear resistance. EN24 steel is primarily used in automotive, machine tool and power generating equipment industries for manufacturing of frames, structural members, fasteners, power transmission shafts, axles, landing gears and etc. This paper mainly focuses on evaluating the performance of various industrial coatings being used in industries for the protection from hydrogen embrittlement. Specimen of EN24 high strength steel specimen were subjected to external hydrogen embrittlement by cathodic pre-charging of specimen in an acidic environment and testing the coated and bare specimen using conventional as well as slow strain rate test technique. This study aims to find out the suitable coatings to mitigate the hydrogen embrittlement of high strength steel. As high strength steel is widely used for manufacturing of fasteners and various critical load taking members the failure due to hydrogen embrittlement could be catastrophic. By use of the suitable coatings on the surface of components one can produce a barrier layer between object and surrounding atmosphere to protect the direct exposure to any electrolytic reaction that resulting in hydrogen embrittlement. The objective of this research work is to evaluate the performance of various coatings to prevent the hydrogen embrittlement.

KEYWORDS: High Strength Steel, External Hydrogen Embrittlement & Coatings

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1. INTRODUCTION

Theory of hydrogen embrittlement was proposed by Johnson [1] long back in 1875. ASTM F2078[2-3] define hydrogen embrittlement as, "An irreversible loss of ductility in a metallic alloys caused due to infusion of hydrogen with combining stresses, whether its internally or externally applied residual stresses". The basic theory and various mechanisms of hydrogen embrittlement were already described in our past work.

This paper discusses about the investigations that were done to evaluate the performance of industrial

coatings to prevent the hydrogen embrittlement of high strength steel namely EN24/AISI 4340 under a condition of tensile load. The behaviour of coating is analysed in terms of its capability to prevent a change in mechanical properties of the base material. The Tensile strength, Yield strength and percentage elongation are measured after exposing the test specimen to cathodic hydrogen that are charging with and without application of coatings for varied duration of time and current density.

Test specimens are subjected to slow strain rate test in order to determine the mechanical properties and time of failure. These observed properties are compared to evaluate the behaviour of coatings under specific condition of hydrogen embrittlement.

A detailed literature review [1-47] summaries Hydrogen Embrittlement (HE) as the hydrogen caused the reduction in the load-bearing and/or the mechanical energy absorption ability of a metallic alloy. Initially, it was found in steels, and now it has been acknowledged that the mechanical properties of most metals and alloys can deteriorate by hydrogen under certain conditions [20]. For instance hydrogen can be absorbed during the metal processing and fabrication, processes as electroplating, solidification, forging and welding. Moreover, hydrogen absorption may occur in specific offshore conditions, such as corrosion and cathodic protection. There are the several methods by which hydrogen can be entered in the material such as cathodic charging, electroplating, during welding etc [21]. From the literature, it was found that if we are performing the cathodic charging then the current density plays an important role in hydrogen absorption in the steel. It was also found that for hydrogen induced phenomena in steel materials, the current density which we are using must be in between 0.02 mA/cm² to 40 mA/cm² [24]. Higher the current density more the chances of hydrogen to be get diffused into the material.

2. PREVENTION OF HYDROGEN EMBRITTLEMENT

Extensive research has been done by scientists & engineers to explore the various ways to prevent hydrogen embrittlement. Some of them are i) addition of alloying elements ii) selection of material with less susceptibility to hydrogen iii) use of barrier layers to prevent hydrogen diffusion iv) change in manufacturing process and application environment v) use of advance coatings to minimize hydrogen penetration etc. Out of all these methods used for controlling hydrogen embrittlement, the use of coatings appears to be more practical, less intricate and economical.

Coatings are being widely used in preventing, minimizing and controlling the effect of an external environment on the metallic and non-metallic substrate. [15]. Coatings creates a barrier film between the parent metal and external environment and then applying a coating protect components from direct exposure however, this might incur an additional cost but proven to be economical for a longer duration when applied on the large volume, thus resulting in the improvement of reliability & reduction in maintenance cycle with lesser chances of failure and enhanced service life [16]. Degradation of base material can be prevented by providing passive [17] or active protection [18]. Passive prevention is done by providing a physical barrier of oxides between object and the surrounding atmosphere [19] hence damage is prevented by mechanically isolating the base material from the aggressive surrounding agents. Active protection is achieved by adding chemicals (inhibitors) to aggressive environments to prevent or minimize the damage. The aim of active corrosion protection is to influence the reactions which took place in the process of material degradation.

Several coatings are being used in industrial applications to protect the components from environmental damage. Authors have listed following coatings on the basis of their experience and further discussion with industry experts for

further experimental analysis to evaluate the performance of coatings in environment promoting hydrogen embrittlement of high strength steel.

- Zinc coating
- Nickel coating
- Nickel-Chrome coating
- Graphene-Zn composite coating

3. SCOPE OF WORK

The scope of research work is to determine the behaviour of coatings when applied on EN24 high strength steel and subject to external hydrogen embrittlement by H-charging using cathodic reaction on standard test specimen of EN24 steel. The coatings behaviour analyzed in this research work shall help scientists, engineers and equipment designers to improve the service life and reliability of structure in performing the desired function.

4. MATERIAL & METHODOLOGY

EN24 Material has been selected for an experimental investigation purpose. EN24 was originally introduced for an use in the motor vehicle and machine tool industries for gears, pinions, shafts, spindles

- It is suitable to produce parts for such as, locomotives, cranes, rolling mills, coal-cutting machinery etc. where the good strength and fatigue resistance is called for.
- Other applications for EN24 are found in die-casting and hot metal working.
- Commercially available material EN24 / AISI 4340 hardened and tempered is investigated to analyze the effect of hydrogen embrittlement on account of zinc electroplating.
- The material has been investigated for two different delivery conditions namely T & X defined by heat treatment cycle and tensile strength.

Chemical and mechanical properties of EN24 are indicated in Table 1 and 2.

Table 1: EN24 Chemical Properties

Carbon	0.35-0.45%	Silicon	0.10-0.35%
Nickel	1.30-1.80%	Manganese	0.45-0.70%
Chromium	0.90-1.40%	Phosphorus	0.05% max
Molybdenum	0.20-0.35%	Sulphur	0.05% max

Table 2: EN24 Mechanical Properties

Grade	U.T.S. (MPa)	Yield (MPa)	Elongation(%)	Impact(KCV) J	Hardness Brinell
T	850-1000	650	13	35	248-302
U	925-1075	855	12	42	269-331
V	1000-1150	750	12	42	293-352
W	1075-1225	940	11	35	311-375
X	1150-1300	1020	10	28	341-401
Y	1225-1375	1095	10	21	363-429
Z	1550	1235	5	9	444

Following methodology is adopted in the present research work on the basis of literature survey.

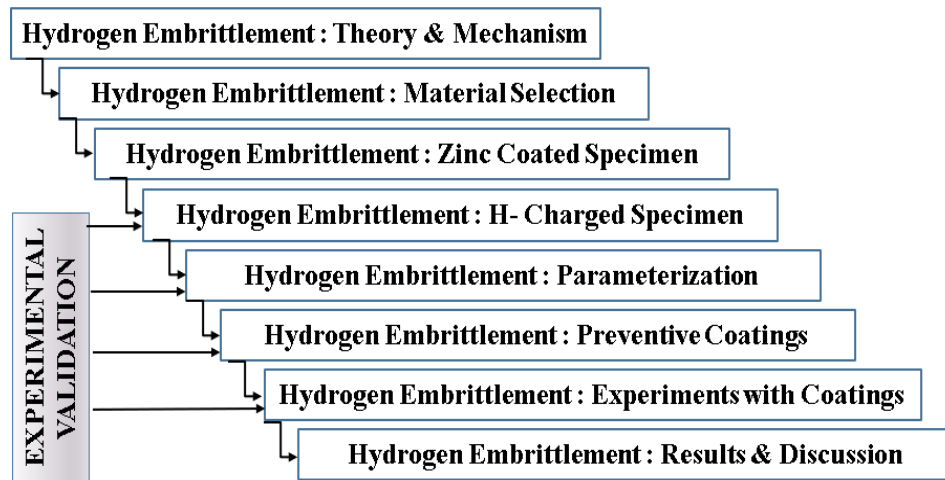


Figure 1: Methodology of Research Work

In this investigation various available industrial coatings were studied vis a vis their behaviour in atmosphere responsible for hydrogen embrittlement by conducting SSRT test on high strength steel specimen after applying coatings and h-charging of specimens. On the basis of results obtained from above experiments, the behaviour of coatings evaluated on the basis of reduction in tensile & yield strength and change in elongation recorded. Finally, EI (embrittlement indices) calculated using time to failure during tensile test and coatings are ranked with the respect to their behaviour in preventing hydrogen embrittlement of high strength steel.

For assessment of HE performance of the material, tensile tests of charged specimen are carried out at controlled strain rate in air. Hydrogen charging of the specimen done by cathodic reaction. During such preparation of solution, a concentrated H_2SO_4 solution is mixed with the water in right proportion. By using this, a dilute H_2SO_4 solution is prepared and cathodic reaction performed for hydrogen diffusion in high strength steel. For cathodic reaction, test specimen is made cathode and platinum wire is used as anode. Hydrogen charging done with current density of 20/40/60 mA/cm^2 for a duration of 4 & 8 hrs for each type of coatings. Charged specimens were tested using slow strain rate test (SSRT) to determine the effect on tensile & yield strength and elongation.

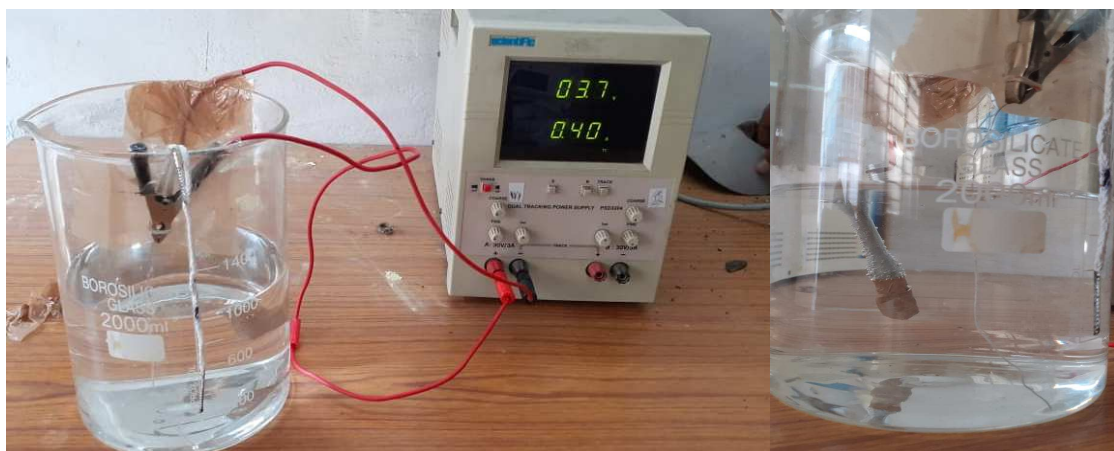


Figure 2: Hydrogen Charging of Test Specimen.



Figure 3: SSRT Flat Specimen.

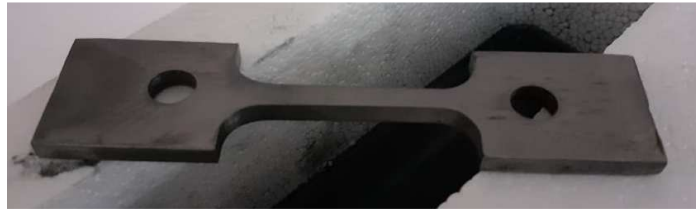


Figure 4: SSRT Round Specimen.



RGo- Zinc Coated Specimen

RGo Propanol Solution



RGo Dip Coated

Figure 5: Coated Specimen for SSRT Test.**Figure 6: Coated SSRT Specimen**

5. RESULTS OF EXPERIMENTS FOR PERFORMANCE EVALUATION OF VARIOUS COATINGS WITH RESPECT TO HYDROGEN EMBRITTLEMENT

Following graphs are plotted using the results of SSRT test indicating the yield strength, tensile strength and % elongation of test specimen coated with various types of coatings.

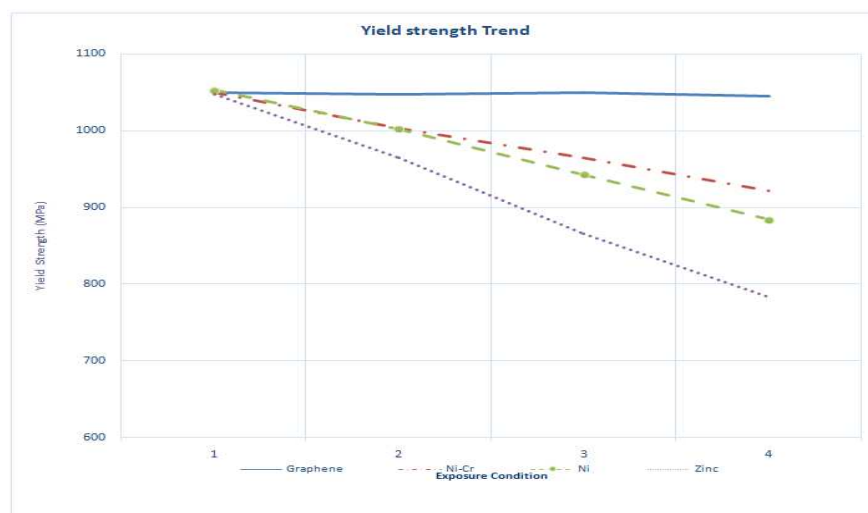


Figure 7: Yield Strength Trends for Various Condition of Charging.

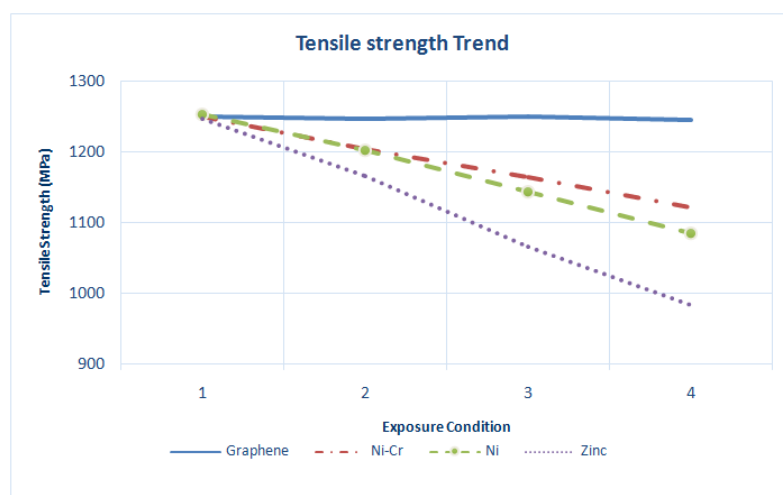


Figure 8: Tensile Strength Trends for Various Condition of Charging.

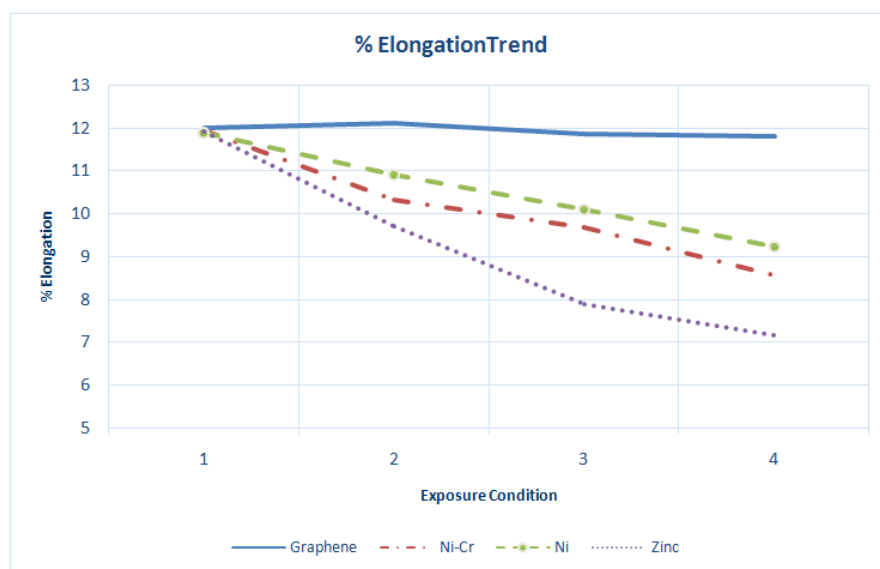


Figure 9: % Elongation Trends for Various Condition of Charging.

5.1 Failure Time Data of Coated Specimen

Following failure time recorded during the testing of various conditions of coated specimen.

Table 3: Failure Time for Coated Specimen

Coating Type`	Failure Time (mins)			
	1	2	3	4
Zinc Electroplating	168	148	123	93
Nickle coating	176	163	145	129
Ni-Cr coating	175	152	129	109
Graphene – Zinc composite coating	181	176	173	171

6. CONCLUSIONS & DISCUSSIONS

- Experiments that performed on selected coatings is to evaluate the behaviour of these coatings in the hydrogen environment and their capability to prevent hydrogen embrittlement, enables to rank these coatings for the use in challenging environment responsible for hydrogen embrittlement.
- Figures 7 to 9 shows that the graphene based zinc coatings are able to completely prevent the hydrogen embrittlement of high strength steel. Test results shows that for different conditions of h-charging the mechanical properties of graphene coated specimen are unchanged. Whereas specimen coated with other types of coatings are subject to hydrogen embrittlement, their tensile and yield strength are decreased by a significant amount.
- Embrittlement indices has been calculated on the basis of failure time as follows –
- The extent of hydrogen embrittlement caused by a particular process is frequently assessed by comparing values of the embrittlement index (EI), which is defined as:-

$EI = 1 - (tp/tc)$, where tp and tc , are the failure time of H charged and uncharged samples, respectively as shown in table 4.

Coatings are ranked as follows on the basis of Embrittlement Indices (EI).

Table 4

Coating Type`	EI
Zinc Electroplating	0.44
Ni-Cr coating	0.37
Nickle coating	0.26
Graphene – Zinc composite coating	0.055

Above indices can be referred as the benchmark while selecting coatings for application like corrosion protection, high pressure hydrogen storage, hydrogen fuel tanks and other conditions leading to hydrogen embrittlement of high strength steel.

7. FUTURE RECOMMENDATIONS AND LIMITATIONS

From this research, further studies suggested are loading conditions, temperature, material type and environmental conditions. Experiments conducted in this research work are specific to EN24 steel used for tensile loading conditions and exposed to H-charging using cathodic reaction. Any change in these conditions requires further experiments to validate the recommendations.

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